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To:	USPTO - Mail Stop Certificate of Corrections Branch	From:	Jason D. Voight
Fax:	(571) 273-8300	Pages:	4
Phone:	(571) 272-1000	Date:	April 28, 2006
Re:	Attorney Docket: PF51063-03	cc:	

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## • Comments:

In re Application of: Christian WULFF et al.

U.S. Patent No.: 6,855,841

Serial No.: 10/664,892

Issue Date: February 15, 2005

Title: PROCESS FOR THE PREPARATION OF  
N-PHOSPHONOMETHYLGLYCINEAttachments: Request for Certificate of Correction  
Certificate of Correction Form PTO/SB/44 (PTO-1050)  
Mark-up copy of Letters Patent

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PATENT

## IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: Christian WULFF et al.

Art Unit: 1626

Patent No.: 6,855,841

Examiner: REI TSANG SHIAO

Issued: February 15, 2005

Confirmation No.: 4266

For: PROCESS FOR THE PREPARATION  
OF N-PHOSPHONOMETHYLGLYCINE

Attorney Docket: PF51063-03

Mail Stop Certificate of Correction  
Commissioner for Patents  
P.O. Box 1450  
Alexandria, VA 22313-1450REQUEST FOR CERTIFICATE OF CORRECTION

Sir:

Applicants herewith submit a Certificate of Correction Form PTO/SB/44. It is respectfully requested that the Certificate of Corrections be entered.

The changes noted on the Certificate of Correction Form PTO/SB/44 correct the errors which occurred on the part of the U.S. Patent and Trademark Office. No fee should therefore be required.

It is not believed that a fee is required for filing of this paper. However, please charge any shortage in fees due in connection with the filing of this paper to Deposit Account No. 14.1437. Please credit any excess fees to such deposit account.

Respectfully submitted,

By:

Jason D. Voight

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Date:

April 28, 2006

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UNITED STATES PATENT AND TRADEMARK OFFICE  
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Page 1 of 1

PATENT NO. : 6,855,841  
APPLICATION NO. : 10/664,892  
ISSUE DATE : February 15, 2005  
INVENTOR(S) : Christian WULFF et al.

It is certified that an error appears or errors appear in the above-identified patent and that said Letters Patent is hereby corrected as shown below:

Claim 1, column 12, line 23, "formulall" should read —formula III—.

## MAILING ADDRESS OF SENDER (Please do not use customer number below):

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## US 6,855,841 B2

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acid and 0.15 mol of dimethylcyclohexylamine in 90 ml of toluene. Stirring is continued for 15 minutes at 0° C. and the mixture is subsequently allowed to come to room temperature. The hydrochloride which has precipitated is filtered through a pressure filter with exclusion of moisture. The tribenzoyl phosphite is characterized via analysis of the filtrate by <sup>1</sup>H NMR and <sup>31</sup>P NMR (yield: 99%). If the residue obtained from the filtrate after the toluene has been distilled off is added to 0.15 mol of 10% strength NaOH, dimethylcyclohexylamine can be recovered quantitatively by phase separation followed by extraction with toluene. The solution is subsequently dried by removing the water azeotropically and can be reused.

## EXAMPLE 11

0.2 mol of sodium benzoate are added to 50 ml of 1,4-dioxane at room temperature with exclusion of moisture. 0.0667 mol of phosphorus trichloride is added dropwise and stirring of the mixture is continued for 20 minutes at 85° C. (colorless suspension). 0.0222 mol of the hexahydrotriazine 1 (X=CN) is added, and stirring of the mixture is continued for another 20 minutes at 85-90° C. (thin suspension, readily stirrable). The dioxane is subsequently distilled off in vacuo at 40° C. 100 ml of concentrated hydrochloric acid are added to the residue and the mixture is refluxed for 4 hours. When cold, the benzoic acid is filtered off and washed (a little cold water). The combined filtrates are extracted twice with in each case 30 ml of toluene, evaporated to dryness on a rotary evaporator and rotary-evaporated three more times with ethanol to remove excess hydrochloric acid. The toluene phase is concentrated and the residue is combined with the benzoic acid which has been recovered.

To isolate the phosphonomethylglycine from the residue of the aqueous phase, this may now be taken up in a little water and precipitated cold at pH 1.0 (addition of NaOH). Complete precipitation is achieved by adding a little methanol, which is recovered from the mother liquor by distillation. Yield: 91%.

The benzoic acid which has been recovered (0.2 mol, purity >99% according to HPLC) is dissolved in 0.2 mol of 5% strength NaOH, the water is subsequently distilled off and the residue is dried. The resulting sodium benzoate together with the dioxane which has been recovered is reused in the synthetic procedure.

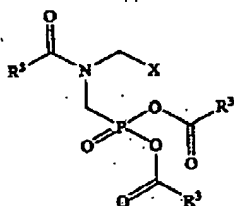
Yield (first recycling): 90%

Yield (second recycling): 84%

Yield (third recycling): 83%.

We claim:

1. A process for the preparation of a phosphono compound of the formula I



in which the radicals R<sup>3</sup>, which can be identical or different, are C<sub>1</sub>-C<sub>18</sub>-alkyl or aryl which is unsubstituted or substituted by C<sub>1</sub>-C<sub>4</sub>-alkyl, NO<sub>2</sub> or OC<sub>1</sub>-C<sub>4</sub>-alkyl, and

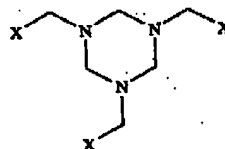
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X is CN, COOZ, CONR<sup>1</sup>R<sup>2</sup> or CH<sub>2</sub>OY, ✓

Y is H or is a radical which is readily exchangeable for H selected from the group consisting of C<sub>1</sub>-C<sub>6</sub>-alkyl, aliphatic aryl having a C<sub>1</sub>-C<sub>6</sub> aliphatic group, and benzoyl;

Z is H, an alkali metal, alkaline earth metal, C<sub>1</sub>-C<sub>18</sub>-alkyl or aryl, which is unsubstituted or substituted by C<sub>1</sub>-C<sub>4</sub>-alkyl, NO<sub>2</sub> or OC<sub>1</sub>-C<sub>4</sub>-alkyl;

R<sup>1</sup> and R<sup>2</sup>, which can be identical or different, are H or C<sub>1</sub>-C<sub>4</sub>-alkyl, in which a hexahydrotriazine derivative of the formula II



is reacted with a triacyl phosphite of the formula III



in which R<sup>3</sup> and X are as defined above.

2. A process as claimed in claim 1, wherein X is CN or COOZ. ✓

3. A process as claimed in claim 1, wherein R<sup>3</sup> is phenyl which is unsubstituted or substituted by C<sub>1</sub>-C<sub>4</sub>-alkyl, NO<sub>2</sub> or OC<sub>1</sub>-C<sub>4</sub>-alkyl, or is CH<sub>3</sub>. ✓

4. A process as claimed in claim 1, which is carried out in an organic solvent.

5. A process as claimed in claim 4, wherein the solvent is dioxane or tetrahydrofuran. ✓

6. A process as claimed in claim 4, wherein a chlorinated organic solvent is used. ✓

7. A process as claimed in claim 6, wherein 1,2-dichloroethane is used as solvent. ✓

8. A process as claimed in claim 1, wherein the compounds of the formulae II and III are employed in essentially equivalent amounts. ✓

9. A process as claimed in claim 1, which further comprises preparing the compound of the formula III by reacting a carboxylic acid of the formula IV



in which R<sup>3</sup> has the meanings stated in claim 3 or a salt thereof with a phosphorus trihalide. ✓

10. A process as claimed in claim 9, wherein an alkali metal salt or the ammonium salt of the carboxylic acid of the formula IV is reacted with the phosphorus halide. ✓

11. A process as claimed in claim 9, wherein the carboxylic acid of the formula IV is reacted with the phosphorus halide in the presence of an amine. ✓

12. A process as claimed in claim 9, wherein the carboxylic acid of the formula IV is reacted with the phosphorus halide in the absence of a base. ✓

13. A process as claimed in claim 9, wherein the reaction is carried out in an inert organic solvent which is selected from among the aromatic or aliphatic hydrocarbons and chlorinated hydrocarbons. ✓

14. A process as claimed in claim 13, wherein the solvent is recovered after the reaction and recycled. ✓